IS 574 (1989): Glassy Sodium Metaphosphate Technical [CHD 1: Inorganic Chemicals]

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Indian Standard

GLASSY SODIUM METAPHOSPHATE — SPECIFICATION

(Fourth Revision)

भारतीय मानक

काँचाभ सोडियम मेटाफॉस्फेट — विशिष्ट

(चौथा पुनरीक्षण)

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Inorganic Chemicals (Misc) Sectional Committee, CDC 3

FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards on 4 September 1989, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

There is a large number of condensed phosphates which are non-crystalline as shown by X-rays. The most interesting compounds in this class are the sodium phosphate glasses which extend from pure P_2O_5 to a composition near a Na_2O/P_2O_5 mole ratio of 1·7. The latter limit occurs because of the increasing tendency of the melt in this region to crystallize upon rapid cooling. The glasses which have been most extensively studied have Na_2O/P_2O_5 mole ratio between 1 and 1·3. The glass with the mole ratio of 1·0 to 1·1 has been called 'Graham's salt', and sometimes 'hexametaphosphate' and other glasses have often been called polyphosphates, with the degree of polymerization given by the proximate analysis. In less precise literature, the term 'hexametaphosphate' has been loosely used for a number of different glasses, not always corresponding to the correct proximate analysis.

Sodium metaphosphate is sometimes produced in the form of glass plates about 1·5 mm thick. It consists of sodium metaphosphate (Graham's salt) with 10 to 15 percent tetrasodium pyrophosphate and has a phosphate content of 67·5 percent (expressed as P_2O_5).

Sodium metaphosphate offers certain technical advantages over other phosphates, like trisodium phosphate and disodium phosphate, in boiler water conditioning, in that it can be passed through most feed lines without the risk of premature deposition of calcium phosphate and it gives lower boiler alkalinities.

Some of the more important commercial applications of phosphates are based on their ability to form soluble complex ions. The term 'sequestration' is commonly used to describe the phenomenon in which soluble complexes prevent the formation of precipitates.

Due to their ability to keep calcium, magnesium and iron salts in solution, metaphoshates are excellent water softeners and detergents. They are particularly useful for leather tanning, dyeing, laundry work, textile scouring and for the 'threshold' treatment of softening industrial water supplies and treatment of cooling water.

This standard was first published in 1954, and subsequently revised in 1961, 1975 and 1980. In this revision, a special requirement on chloride for use in boiler water has been incorporated. Two methods of test for chloride estimation have been specified, namely, volumetric and turbidimetric.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

GLASSY SODIUM METAPHOSPHATE — SPECIFICATION

(Fourth Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for glassy sodium metaphosphate, technical $[(NaPO_3)]_n$ with Na_2O/P_2O_5 mole ratio of 1·0 to 1·1. It is also known as sodium hexametaphosphate or Graham's salt.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to the standard:

IS No.	*Title*
265 : 1987	Specification for hydrochloric acid (*third revision*)
915 : 1975	Specification for one-mark volumetric flask (*first revision*)
1070 : 1977	Specification for water for general laboratory use (*second revision*)
4905 : 1968	Methods for random sampling

3 GRADES

The material shall be of the following two grades:

a) Grade 1, and

b) Grade 2.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of powder, flakes and as small broken glass-like particles.

4.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A.

4.3 Special Requirement for Use in Boiler Water

When the material is to be used as corrosion inhibitor in coolant water system, the chloride content (as Cl) shall be 0·03 percent by mass, *Max* when tested as per the method prescribed in Annex B.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

5.2 Marking

The packages shall be securely closed and marked legibly and indelibly with the following information:

a) Name and grade of the material;

b) Name of the manfacturer, his recognized trade-mark, if any;

c) Net mass of the material in the package;

d) Date of manufacture; and

e) Batch number.

6 SAMPLING

The method of drawing representative samples

Table 1 Requirements for Glassy Sodium Metaphosphate, Technical

Sl No.	Characteristic	Requirement		Method of Test, Ref Cl No. in Annex A
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Matter insoluble in water, percent by mass, *Max*	0·20	0·50	A-2
ii)	Soluble iron compounds (as Fe), percent by mass, *Max*	0·03	0·035	A-3
iii)	Phosphates (as P_2O_5), percent by mass, *Min*	67·0	62·0	A-4
iv)	Loss on ignition, percent by mass, *Max*	3·0	5·0	A-5
v)	Sequestering power	To pass test	To pass test	A-6
vi)	Orthophosphate content, percent by mass, *Max*	1·5	1·5	A-7

of the material, the number of tests to be performed and the method of finding out the conformity of the material to the requirements of this standard shall be as prescribed in Annex C.

ANNEX A

(*Clause* 4.2)

METHODS OF TEST FOR GLASSY SODIUM METAPHOSPHATE

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 MATTER INSOLUBLE IN WATER

A-2.1 Procedure

Weigh accurately about 10 g of the material and dissolve in about 150 ml of water. Filter the residue, if any, through a tared filter paper or a sintered glass crucible (G. No. 4) or a Gooch crucible. Wash the residue thoroughly with water till it is free from all soluble compounds and dry to constant mass at 105 to 110°C. Collect the filtrate and the washings in a 250-ml volumetric flask and make up the volume with water. Preserve it for test under **A-6**.

A-2.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = mass in g of the residue obtained, and

M = mass in g of the material taken for the test.

A-3 SOLUBLE IRON COMPOUNDS

A-3.0 Outline of the Method

Iron is determined colorimetrically by measuring the transmittance of reddish violet colour produced by ferric and ferrous iron with thioglycollic acid at *p*H 10.

A-3.1 Apparatus

A-3.1.1 *Photoelectric Absorptiometer*

A-3.1.2 *One-Mark Graduated Flasks*

100 ml capacity (*see* IS 915 : 1975).

A-3.2 Reagents

A-3.2.1 *Dilute Hydrochloric Acid*

Approximately 5 N.

A-3.2.2 *Sodium Citrate Solution*

1 M.

Dissolve 29·4 g of sodium citrate in 100 ml of water; or

Ammonium Citrate Solution

Dissolve 43·0 g of ammonium citrate in 100 ml of water.

A-3.2.3 *Ammonium Hydroxide*

Approximately 10 N.

A-3.2.4 *Thioglycollate Reagent*

Add 20 ml of ammonium hydroxide to 30 ml of water, then add a mixture of 10 ml of thioglycollic acid and 40 ml of water.

A-3.2.5 *Standard Iron Solution*

Dissolve 0·702 g of ferrous ammonium sulphate [$Fe(NH_4)_2SO_4.6H_2O$] in 10 ml of dilute sulphuric acid (10 percent, *v/v*) and dilute with water to 1 000 ml. Take 10 ml of this solution and dilute to 100 ml. One millilitre of this solution contains 0·01 mg of iron (as Fe).

A-3.3 Procedure

Take several aliquots of the standard iron solution in one-mark graduated flasks, add to each of these flasks 4 ml of sodium citrate solution or 2 ml of ammonium citrate solution, 5 ml of thioglycollate reagent and 15 ml of ammonium hydroxide, swirling after each addition. Dilute to the mark and mix well. After 10 minutes determine the absorbance against a reagent blank at 535 nm using green filter No. 4 and 4-cm cell (use 1 cm cell if the colour is too strong). Draw a calibration curve by plotting concentration (in μg/ml) against absorbance.

A-3.3.1 Weigh accurately about 1 g of the material and dissolve in 20 ml of water. Add 5 ml of dilute hydrochloric acid. Stir slowly with glass rod, cover with watch-glass and boil gently

for 15 to 20 minutes, cool and transfer to a one-mark graduated flask. Add 4 ml of sodium citrate solution or 2 ml of ammonium citrate solution, 5 ml of thioglycollate reagent and 15 ml of ammonium hydroxide. Dilute to the mark and after 10 minutes determine the absorbance at 535 nm using green filter No. 4 and 4-cm cells (use 1-cm cell if the colour is too strong). Read the concentration (in μg/ml) of this solution from the calibration curve and calculate the total amount of soluble iron present (as Fe).

A-4 PHOSPHATE CONTENT

A-4.0 Outline of the Method

The material is dissolved in waster, if necessary, with a little hydrochloric acid. Phosphorus is precipitated as quinoline phosphomolybdate. The precipitate is filtered under mild suction on a thick pad of filter paper pulp, washed with water and dissolved in excess of standard alkali. The unreacted alkali is back titrated against standard hydrochloric acid.

A-4.1 Reagents

A-4.1.1 *Concentrated Hydrochloric Acid*

See IS 265 : 1987.

A-4.1.2 *Sodium Hydroxide Solution*

10 percent (*m/v*).

A-4.1.3 *Citric Acid*

Solid.

A-4.1.4 *Citro-molybdate Reagent*

Stir 54 g of molybdic acid, anhydride (MoO_3) with 200 ml of water and 11 g of sodium hydroxide while heating until molybdic acid, anhydride dissolves. Add sodium hydroxide solution gradually. This will take 20 to 30 minutes. Dissolve 120 g of pure citric acid in 250 to 300 ml of water and add 140 ml of concentrated hydrochloric acid. Pour the molybdate solution to the acid solution with stirring, cool and filter through a pulp pad, if necessary. Dilute to 1 litre. The solution will be blue or green in colour. Add drop-wise a dilute (0·5 to 1·0 percent) solution of potassium bromate until the blue colour is discharged. The solution is stable if kept in the dark.

A-4.1.5 *Quinoline Hydrochloride Solution*

Take 60 ml of concentrated hydrochloric acid and 300 to 400 ml of water in a 1-litre beaker and warm it to 70 to 80°C. Pour 50 ml of purified quinoline (free from reducing agents) in a thin stream into the dilute acid while stirring. When quinoline has dissolved, cool the solution, filter through a pulp and dilute to 1 litre.

NOTE — Synthetic quinoline is usually suitable, but preferably purified by dissolving in hydrochloric acid and precipitating the double chloride [$(C_9H_7N)_2$. $ZnCl_4$] with excess of zinc chloride dissolved in dilute hydrochloric acid. Quinoline is regenerated with excess of sodium hydroxide solution and distilled.

A-4.1.6 *Sodium Hydroxide Solution*

0·1 N approximately.

A-4.1.7 *Standard Hydrochloric Acid*

0·1 N.

A-4.1.8 *Mixed Indicator Solution*

3 volumes of 0·1 percent thymol blue mixed with 2 volumes of 0·1 percent phenolphthalein in 60 percent of ethyl alcohol.

A-4.2 Procedure

A-4.2.1 Weigh about 0·5 g of the material in a 250-ml beaker. Add 100 ml of water and 10 ml of concentrated hydrochloric acid, warm on a hot-plate until dissolution is complete and boil gently for 45 to 60 minutes to ensure that hydrolysis of the condensed phosphate takes place. Filter to remove any insolubles present. Transfer to a 250-ml measuring flask and make up to the mark.

A-4.2.2 Take aliquot equivalent to 0·05 to 0·10 g of the sample in a 500-ml conical flask. Neutralize with 10 percent sodium hydroxide solution and add 2 to 3 ml excess of concentrated hydrochloric acid. Make the volume to about 120 ml by adding water. Add 1 g of citric acid, heat to boiling and add from a burette in thin stream, 20 ml of quinoline hydrochloride solution. Put a rubber stopper and shake vigorously for about 5 minutes. Allow the precipitate to settle down and the solution to cool.

A-4.2.3 Filter under suction through a thick pad of filter pulp prepared on a perforated disc in a funnel. Wash the precipitate and the flask with water till free from acid (about 10 ml of the washing and 4 drops of the indicator should show colour change from voilet to yellow with 1 drop of 0·1 N sodium hydroxide solution). Transfer the pad of filter paper back with the precipitate to the original flask. Add 50 to 60 ml of water and then add 0·1 N sodium hydroxide solution with stirring in multiples of 25 ml from a pipette till the precipitate dissolves completely. Add 5 to 6 drops of the indicator solution and titrate the excess of alkali with standard hydrochloric acid till violet colour changes to yellow. The end point is sharp.

A-4.2.4 Run a blank determination with the same quantity of sodium hydroxide solution which was added in the test and titrate with standard hydrochloric acid using 5 to 6 drops of indicator solution.

A-4.3 Calculation

$$\text{Phosphates (as } P_2O_5 \text{), percent by mass} = \frac{(V_1 - V_2)\, N \times 0.2732}{M}$$

where

V_1 = volume in ml of standard hydroch oric acid required for the blank test,

V_2 = volume in ml of standard hydrochloric acid required for the sample,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for the test.

A-5 LOSS ON IGNITION

A-5.1 Procedure

Weigh accurately about 1 g of the material and heat it in a tared platinum crucible until it melts. Keep it molten for 5 minutes. Cool in a desiccator and weigh.

A-5.2 Calculation

$$\text{Loss on ignition, percent by mass} = 100 \times \frac{M - M_1}{M}$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material after ignition.

A-6 SEQUESTERING POWER

A-6.1 Prepared Sample Solution

Take exactly 200 ml of the solution reserved in **A-2.1** in a 1 000 ml volumetric flask and make up the volume to the mark with water.

A-6.2 Reagents

A-6.2.1 *Calcium Nitrate Solution*

Dissolve 0·589 g of calcium nitrate [$Ca(NO_3)_2 . 4H_2O$] in water and dilute to 1 000 ml.

A-6.2.2 *Phenolphthalein Indicator Solution*

0·1 percent in 60 percent reotified spirit.

A-6.2.3 *Dilute Sodium Hydroxide Solution*

Approximately 0·1 N.

A-6.2.4 *Dilute Sulphuric Acid*

Approximately 0·1 N.

A-6.2.5 *Dilute Hydrochloric Acid*

Approximately 5 N.

A-6.2.6 *Standard Calcium Chloride Solution*

Weigh accurately 0·20 g of calcium carbonate in a chemically-resistant glass vessel and dissolve it in dilute hydrochloric acid taking care to keep the vessel covered with a clock-glass to prevent loss by spurting. When all the solid has dissolved, evaporate to dryness on the water-bath, add a a little water and again evaporate to dryness. Repeat the evaporation several times to ensure complete expulsion of the free acid. Finally dissolve the residue in water and make up to 1 000 ml in a volumetric flask. 50 ml of this solution corresponds to 10 mg of calcium carbonate.

A-6.2.7 *Alcoholic Potassium Hydroxide Solution*

Dissolve 20 g of potassium hydroxide in 180 ml of rectified spirit.

A-6.2.8 *Standard Soap Solution*

Weigh about 50 g of oleic acid into a beaker and add 100 ml of alcoholic potassium hydroxide solution. Continue adding this solution from a burette until a drop of the oleate just gives a red colour with phenolphthalein spotted on a white plate (about 10 ml being required for this). Make the volume up to 1 000 ml by addition of rectified spirit. Set aside the solution in a hot plate for 24 hours. Filter through a double filter paper and standardize against standard calcium chloride solution as described below.

A-6.2.8.1 *Standardization of the soap solution*

Pipette out 50 ml of standard calcium chloride solution into a glass-stoppered bottle of about 250 ml capacity. Run in 1 ml of the soap solution from a burette into the bottle, close the bottle and shake it vigorously for a short time. If no permanent lather is formed, add another 1 ml and shake again.

Continue the addition of the soap solution, shaking the mixture well after each addition until a lather is produced which remains for a short time when the bottle is laid upon the bench. The titration is complete when the lather remains upon the surface in an unbroken layer for 5 minutes even when the bottle is rolled half-way round on its side. Towards the end of the titration, the volume of soap solution which is added each time should be decreased and it should finally not exceed 0·2 to 0·3 ml. Repeat the titration with another 50 ml of the standard calcium chloride solution, adding the soap solution 1 ml (or less) at a time, with shaking after each addition. Dilute the standardized soap solution with calculated volume of rectified spirit so that exactly 14·25 ml of the final solution are required to produce a lather with 50 ml of standard calcium chloride solution.

A-6.3 Procedure

Transfer 12·5 ml of the prepared sample solution (*see* **A-6.1**) into a stoppered 250-ml bottle. Add to it 50 ml of calcium nitrate solution together with few drops of phenolphthalein indicator. Add dilute sodium hydroxide solution or dilute sulphuric acid as the case may be, until the colour of the solution is very faint pink. Then add standard soap solution until a lather, permanent for 5 minutes, is obtained on shaking for 30 seconds.

A-6.3.1 The material shall be regarded to have satisfied the requirements of the test if the volume of standard soap solution used is equal to or less than that required to produce a permanent lather with 62·5 ml of distilled and boiled water.

A-7 ORTHOPHOSPHATE CONTENT

A-7.0 Outline of the Method

Unconverted orthophosphate content of the material is determined colorimetrically.

A-7.1 Apparatus

A-7.1.1 *Photoelectric Absorptiometer*

A-7.2 Reagents

A-7.2.1 *Standard Phosphate Solution*

Dry potassium bihydrogen phosphate crystals at 105 to 110°C for 1 hour. Weigh 0·963 8 g of dry salt and dissolve in 500-ml measuring flask and raise the volume up to the mark. One millilitre of this solution is equivalent to 1·0 mg of P_2O_5.

A-7.2.2 *Ammonium Metavanadate*

A-7.2.2.1 Dissolve 1·0 g of ammonium metavanadate in hot water in a beaker. Allow it to cool (solution *A*).

A-7.2.2.2 Dissolve 20 g of ammonium molybdate in hot water in a beaker. Allow it to cool (solution *B*).

A-7.2.2.3 Transfer solution *A* to a 1-litre measuring flask, add 225 ml of perchloric acid with constant shaking. Then add solution *B* to the flask with constant shaking and raise the volume up to the mark.

A-7.3 Procedure

A-7.3.1 Weigh about 1 g of the sample and dissolve it in sufficient amount of water. Transfer it to 100 ml measuring flask and make up the volume by water (*solution C*). Take between 2 to 25 ml of freshly prepared solution *C* (to avoid hydrolysis and to get absorbance reading more than 0·10 depending upon whether orthophosphate content is high or low) in a 100 ml measuring flask and add to it about 35 ml of water and 25 ml of ammonium metavanadate reagent and make up the volume to the mark. Measure the absorbance at 420 nm after 5, 10 and 15 minutes have passed exactly after addition of ammonium metavanadate reagent. If R_1, R_2 and R_3 are absorbances after 5, 10 and 15 minutes then absorbance due to P_2O_5 content in orthophosphate form before hydrolysis is given by:

$$R = R_1 - \frac{[(R_2 - R_1) + (R_3 - R_2)]}{2}$$

A-7.3.2 *Preparation of Calibration Curve*

Take 0, 0·5, 1·0, 1·5, 2·0 and 2·5 ml of standard phosphate solution in a series of 100-ml measuring flasks. Add to it about 35 ml of water and 25 ml of ammonium metavanadate reagent, make up to the mark and shake. Measure the absorbance of each solution at 420 nm. Plot absorbance against concentration to get a standard graph. Find out the P_2O_5 concentration (in mg) of unknown sample for absorbance *R* from the standard graph.

A-7.4 Calculation

$$\text{Orthophosphate content (as } P_2O_5\text{), percent by mass} = \frac{M}{1\,000} \times \frac{100}{V} \times \frac{100}{M_1}$$

where

M = orthophosphate content in g in 100 ml of the sample solution,

M_1 = mass in g of the material taken for the test, and

V = aliquot of the solution taken.

ANNEX B

(*Clause* 4.3)

METHOD OF TEST FOR CHLORIDE CONTENT

B-1 CHLORIDE CONTENT

B-1.0 Two methods have been specified, namely, turbidimetric and volumetric. Turbidimetric method shall be the routine method and volumetric method shall be referee method.

B-1.1 Turbidimetric Method

B-1.1.1 *Reagents*

B-1.1.1.1 *Dilute nitric acid*

1:9 (*v/v*).

B-1.1.1.2 *Silver nitrate solution*

10 percent (*m/v*).

B-1.1.1.3 *Standard chloride solution*

Dissolve 1·648 4 g of pure and dry sodium chloride in water and dilute to 1 litre. Dilute 10 ml of this

solution to 1 000 ml so that one ml contains 0·01 mg of chloride (as Cl).

B-1.1.2 *Apparatus*

B-1.1.2.1 *Nessler cylinder*

B-1.1.3 *Procedure*

Dissolve 1 g of the sample in water and dilute to 100 ml in a volumetric flask. To 10 ml of this solution taken in a 50 ml Nessler cylinder add 10 ml dilute nitric acid followed by two drops of silver nitrate solution. Dilute to the mark with water and mix well. Any turbidity produced should not be greater than that produced in a control where 3 ml of standard chloride containing 0·01 mg of Cl per ml was treated in a similar manner.

B-1.2 Volumetric Method

B-1.2.1 *Reagents*

B-1.2.1.1 *Aluminium hydroxide suspension*

Dissolve 125 g of potassium or ammonium alum in 1 litre of water. Precipitate the aluminium by adding ammonium hydroxide slowly and with stirring. Wash the precipitate by successive decantation with several portions of water until free from sulphates.

B-1.2.1.2 *Hydrogen peroxide*

30 percent.

B-1.2.1.3 *Calcium carbonate*

B-1.2.1.4 *Standard nitric acid*

0·1 N.

B-1.2.1.5 *Potassium chromate solution*

Dissolve 5 g of potassium chromate in water and make up to 100 ml. Add silver nitrate solution to produce a slight red precipitate and filter.

B-1.2.1.6 *Standard silver nitrate solution*

Dissolve 4·791 g of silver nitrate, dried at 105 ± 2°C, in water and make up to 1 000 ml. One millilitre of the diluted solution is equivalent to 1 mg of chlorides (as Cl). Standardize with a standard chloride solution. The solution shall be kept in the dark.

B-1.2.2 *Procedure*

B-1.2.2.1 Dissolve 5 g of the sample in water in a 100-ml volumetric flask filter if necessary and dilute to the mark. Use this solution for the titration. If the sample is coloured, decolourize by adding 3 ml of aluminium hydroxide suspension. Stir thoroughly and after a few minutes filter and wash with 10 to 15 ml of water. If sulphites are present, add 1 ml of hydrogen peroxide with stirring.

B-1.2.2.2 Place the sample treated as in **B-1.2.2.1** in a porcelain basin. If the *p*H of the sample is less than 6·8, add a small amount of calcium carbonate to the sample in the basin so as to neutralize the acidity. If the *p*H is above 10, determine the amount of standard nitric acid required to neutralize 100 ml of the sample, and add this amount of the acid to the portion used for the chloride determination, and then add a trace of calcium carbonate. Add 1 ml of potassium chromate solution and titrate with standard silver nitrate solution with constant stirring until there is perceptible reddish colouration. Subtract 0·2 ml from the titration figure to allow for the excess of reagent required to form silver chromate.

NOTE — If the sample requires more than 25 ml of silver nitrate solution, repeat the determination with a smaller quantity of the sample diluted to 100 ml with water. If chlorides are present in very small quantities, concentrate 500 or 1 000 ml in a porcelain dish to 100 ml, rub down sides of the dish carefully, neutralize as above and titrate with silver nitrate solution.

B-1.2.3 *Calculation*

$$\text{Chlorides (as Cl), percent by mass} = \frac{V_1 \times f}{M \times 10}$$

where

V_1 = volume in ml of standard silver nitrate solution used in the titration,

f = mg of chloride (as Cl) equivalent to 1 ml of silver nitrate solution, and

M = mass in g of the sample taken for the test.

ANNEX C

(*Clause* 6)

SAMPLING OF SODIUM METAPHOSPHATE, TECHNICAL

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

C-1.1 Samples shall not be taken at a place exposed to the adverse effects of weather.

C-1.2 The sampling instruments and sample containers shall be clean and dry.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

C-1.5 The samples shall be placed in suitable, clean, dry, and airtight glass or other suitable containers on which the material has no action.

C-1.6 The sample containers shall be of such sizes that they are almost completely filled by the sample.

C-1.7 Each sample container shall be suitably stoppered and sealed airtight after filling, and marked with full particulars of the material (*see* **5.2**) and the date of sampling.

C-1.8 Samples shall be stored in a cool and dry place.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

C-2.1.1 Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of the specification.

C-2.2 The number (n) of containers to be selected at random from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected from Lots of Different Size

(*Clause* C-2.2)

Lot Size (N)	Number of Containers to be Selected (n)
(1)	(2)
Up to 15	3
16 ,, 50	4
51 ,, 100	5
101 ,, 300	7
301 ,, 500	8
501 ,, 1 000	9
1 001 and above	10

C-2.3 These containers shall be chosen at random from the lot. For random selection procedures, guidance can be had from IS 4905 : 1968.

C-3 TEST SAMPLES AND REFEREE SAMPLES

C-3.1 From each of the containers selected according to **C-2.3**, draw with an appropriate sampling instrument a representative portion of the material sufficient for carrying out the tests specified under **4**. These shall constitute the individual samples.

C-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

C-3.3 The material in the individual and composite test samples shall be divided into three equal parts and transferred to separate bottles, sealed and labelled with full identification particulars of the samples (*see* **C-1.7**). The material in each such bottle shall constitute a test sample. Separate these test samples representing individual and the composite samples into three identical sets of test samples.

C-3.4 Distribution of Test Samples

One set of test samples shall be sent to the purchaser and another to the supplier. The third set of test samples bearing the seals of the purchaser and the supplier shall constitute the referee sample to be used in case of dispute between the purchaser and the supplier and shall be kept at a place agreed to between the two.

C-4 NUMBER OF TESTS

C-4.1 Tests for determination of matter insoluble in water and phosphate shall be carried out on

each of the set of the test samples representing individual samples.

C-4.2 Tests for determination of the remaining characteristics shall be performed on the composite sample.

C-5 CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples

For those characteristics which are tested on individual samples, the mean and range of test results shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of the individual test results}}{\text{Number of test results}}$$

$$\text{Range } (R) = \text{Difference between the maximum and minimum values of test results}$$

C-5.1.1 For declaring the conformity of the lot to the specified requirement of matter insoluble in water, $\bar{X} + 0 \cdot 6\ R$ computed from test results shall be less than or equal to the relevant limit prescribed in Table 1. The conformity criterion for phosphates shall be that ($\bar{X} - 0 \cdot 6\ R$) computed from test results shall be greater than or equal to relevant limit prescribed in Table 1.

C-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all the characteristics tested on the composite sample (*see* **C-4.2**), the test results shall comply with the corresponding specified values.

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 FEBRUARY 1994
TO
IS 574 : 1989 GLASSY SODIUM METAPHOSPHATE — SPECIFICATION

(Fourth Revision)

(*Page* 3, *clause* A-4.2.2) — Substitute the following for the existing clause:

A-4.2.2 Take aliquot equivalent to 0·03 to 0·05 g of the sample in a 500-ml conical flask. Neutralize with 10 percent sodium hydroxide solution and add 2 to 3 ml excess of concentrated hydrochloric acid. Make the volume to about 120 ml by adding water. Add 1 g of citric acid followed by 60 ml of citromolybdate reagent, heat to boil and add slowly while boiling, 40 ml of quinoline hydrochloride solution from a burette in thin stream with agitation. Boil for further 5 to 10 minutes with agitation. Put a rubber stopper and shake vigorously for about 5 minutes. Allow the precipitate to settle down and the solution to cool.'

(*Page* 3, *clause* A-4.2.3, *line* 6) — Substitute 'yellow to violet' *for* 'violet to yellow'.

(*Page* 5, *clause* A-7.4, *line* 5) — Substitute '*M* = mass in mg of phosphorous pentoxide in the aliquot' *for* '*M* = orthophosphate content in g in 100 ml of the sample solution'.

(CHD 003)

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